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STUDY OF PHASE FORMATION IN $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ NANOCOMPOSITES AS A RESULT OF THERMAL ANNEALING

The aim of this work is systematic study of the thermal annealing effect on the preparation of nanostructured composites $\text{NdFeO}_3/\text{Fe}_2\text{O}_3$ with a spinel type structure. The interest in these nanocomposites is due to the enormous potential of their application as a basis for magnetic devices, catalysts, and magnetic carriers for targeted drug delivery. As a synthesis method, two-stage synthesis was used, which includes mechanochemical grinding of nanopowders Fe_2O_3 and Nd_2O_3 in a planetary mill, followed by thermal annealing of the resulting mixture in a wide temperature range: 600-1000°C. During the studies carried out, it was found that in the initial state the obtained nanocomposites are a mixture of a solid solution of interstitial and substitutional Fe_2O_3 and Nd_2O_3 . At an annealing temperature of 600°C, the onset of the formation of the NdFeO_3 phase is observed, which at a temperature of 1000°C is fully formed and dominates in the composite structure (content more than 85%). It was also found that during thermal sintering, the processes of phase transformations of the $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ type are accompanied by an increase in the particle size by a factor of 1.5-2.

Key words: spinel, $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$, nanocomposites, catalysts, thermal annealing, phase formation.

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$\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ нанокөпозиттеріндегі термиялық өңдеудің нәтижесінде фазалық құрылуды зерттеу

Бұл жұмыстың мақсаты – шпинель типті құрылымы бар $\text{NdFeO}_3 / \text{Fe}_2\text{O}_3$ нано құрылымды композиттерді алуға термиялық өңдеудің әсерін жүйелі түрде зерттеу болып табылады. Бұл нанокөпозиттерге деген қызығушылық оларды магниттік құрылғылардың, катализаторлардың және дәрі-дәрмектерді мақсатты түрде жеткізуге арналған магниттік тасымалдағыштардың негізі ретінде қолданудың үлкен әлеуетіне байланысты. Бұл жұмысты синтез әдісі ретінде екі сатылы синтез қолданылды, оның ішінде Fe_2O_3 және Nd_2O_3 нано ұнтақтарын планетарлық диірменде механохимиялық ұнтақтау, содан кейін алынған қоспаны кең температуралық диапазонында: 600 – 1000 °С – термиялық күйдіру өткізілді. Жүргізілген зерттеулер барысында бастапқы күйде алынған нано композиттер – Fe_2O_3 және Nd_2O_3 ену және алмастырғыш қатты ерітінді қоспасы түрінде болатыны анықталды. Және де өңдеу температурасы 600 °С кезінде NdFeO_3 фазасының түзілуінің басталуы байқалып, ал 1000 °С температурада композиттің құрылымында толық қалыптасады және үстемдік ететіні (құрамы 85 %-дан жоғары) байқалды. Сондай-ақ, термиялық қақтау кезінде $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ типті фазалық түрлену процестері бөлшектердің өлшемдерінің 1,5 – 2 есе ұлғаюымен қатар жүретіндігі анықталды.

Түйін сөздер: шпинель, $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$, нанокөпозиттер, катализаторлар, термиялық өңдеу, фазалық құрылу.

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Изучение фазообразования в нанокompозитах $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ в результате термического отжига

Целью данной работы является систематическое изучение влияния термического отжига на получение наноструктурных композитов $\text{NdFeO}_3/\text{Fe}_2\text{O}_3$ со шпинельного типа структурой. Интерес к данным нанокompозитам обусловлен огромным потенциалом их применения в качестве основы для магнитных устройств и катализаторов, а также и магнитных носителей для адресной доставки различных лекарств. В качестве метода синтеза в работе использовался двухэтапный синтез, включающий в себя механохимическое перемалывание нанопорошков Fe_2O_3 и Nd_2O_3 в планетарной мельнице, далее с последующим термическим отжигом полученной смеси в широком диапазоне температур 600 – 1000 °С. В ходе проведенных исследований установлено, что в исходном состоянии полученные нанокompозиты представляют собой смесь твердого раствора внедрения и замещения Fe_2O_3 и Nd_2O_3 . При температуре отжига 600 °С наблюдается начало формирования фазы NdFeO_3 , которая при температуре 1000 °С является полностью сформированной и доминирует в структуре композита (содержание более 85 %). Также установлено, что при термическом спекании, процессы фазовых превращений типа $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ сопровождаются увеличением размеров частиц в 1,5-2 раза.

Ключевые слова: шпинель, $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$, нанокompозиты, катализаторы, термический отжиг, фазообразование.

Introduction

One of the promising areas of research in modern materials science is the study of the kinetics of phase transformations in nanostructured materials under the influence of external factors [1-3]. Interest in this area is due to the need to obtain new data on the processes of phase formation in nanomaterials, which have significant differences from bulk analogs, and can also be accompanied not only by phase transformations and changes in structural characteristics, but also by significant changes in morphological parameters, consisting in a change in the shape of nanoparticles. their density and porosity. At the same time, in the case of nanostructured materials, in most cases, the formation of new phases occurs under significantly different conditions from similar massive samples [4,5]. This is primarily due to small particle sizes, which lead to the fact that a large number of structural metastable distortions and deformations occur in the structure, which under external factors can lead to an acceleration of phase transformation processes. In this regard, the study of such processes, as well as obtaining new data on the dynamics of phase transformations, as well as the possibility of obtaining nanocomposite structures is of great interest not only from a fundamental point of view, but also of great potential in industrial application [6-10].

Among the wide variety of magnetic nanostructures, structures based on iron oxide doped

with other magnetic components, such as nickel, cobalt, or neodymium, are of particular interest [11-15]. Interest in these types of structures is due to their unique magnetic properties, which, as is known, directly depend on the structural characteristics, including the degree of ordering, and phase composition. Also of interest are spinel-type structures of the ABO_3 type, where A is the magnetic component of the dopant and B is iron [16-20]. Such structures have great potential for application not only in biomedicine, as a basis for magnetic resonance spectroscopy, targeted drug delivery, but also in catalysis, purification of aqueous media, magnetic sensors, etc. In this case, the choice of neodymium as component A in most cases plays an important role both in the magnetic performance of nanoparticles and in the increase in the adsorption capacity of nanocomposites, which are increasingly used as adsorbents or as a basis for catalysts for the purification and disposal of harmful compounds and heavy metals from aqueous media. [21-25].

Summarizing all of the above, this study aimed at studying the dynamics of phase transformations in nanocomposites $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$, the ultimate goal of which is to obtain spinel-type NdFeO_3 structures, is one of the topical trends in the development of nanomaterials science, and the data obtained on phase transformations and methods of obtaining nanostructures NdFeO_3 can later be used for semi-industrial applications.

Experimental part

To obtain NdFeO₃/Fe₂O₃ nanocomposites, a two-stage synthesis technique was used, including the mechanochemical mixing of two Fe₂O₃ and Nd₂O₃ powders in a planetary mill for 1 hour at a grinding speed of 400 rpm. The grinding temperature mode did not exceed 50°C, in order to avoid the initiation of phase transformation processes during mixing. The second stage consisted in isochronous thermal annealing of the resulting mixture in the temperature range of 600-1000°C in the atmosphere for 5 hours, followed by cooling together with the furnace for 24 hours. After annealing, the resulting nanocomposites were placed in plastic containers to avoid oxidation and air degradation processes.

The assessment of morphological features was carried out using scanning electron microscopy implemented using scanning electron microscopy Jeol 7500F.

The dynamics of nanocomposites phase transformations was studied on a D8 Advance ECO powder diffractometer (Bruker). X-ray diffraction patterns were recorded in the Bragg-Brentano geometry, in the angular range $2\theta=20-80^\circ$ with a step of 0.03° , Cu-K $\lambda=1.54 \text{ \AA}$.

Results and discussion

Figure 1 shows the results of a study of the morphological features of synthesized nanocomposites, depending on the annealing temperature. As can be seen from the presented SEM images of the initial nanocomposites, after grinding, the structure of the composites is represented by particles of various irregular shapes, covered with a thin porous layer, the presence of which is caused by the processes of grinding and rubbing. In turn, annealing at a temperature of 600°C leads to the formation of stuck together fused agglomerates of particles, the size of which varies from several hundred nanometers to several microns. The agglomerates themselves consist of particles, the size of which varies from 10 to 30 nm. Further annealing of the samples at a temperature of 800°C leads to the formation of spherical or sphere-like particles, the size of which varies from 20 to 50 nm. At a temperature of 1000°C, an increase in particle sizes to 50-70 nm is observed, with the formation of large dendritic-type clusters. In this case, in contrast to the samples in the initial state, the nanoparticles obtained at temperatures of 800°C and 1000°C have a denser structure, without visible cracks or porous inclusions, which indicates

a high degree of structural ordering and an increase in the strength properties of nanocomposites.

The assessment of structural changes, as well as the dynamics of phase transformations as a result of thermal annealing, was carried out using the method of X-ray phase analysis, the results of which are shown in Figure 2. The general view of the obtained diffraction patterns indicates a polycrystalline structure of the synthesized structures, and the low intensity of reflections and their broadened shape indicate nanoscale dimensions of X-ray diffraction patterns. At the same time, the general appearance of changes in diffraction patterns depending on the annealing temperature, characterized by the formation of new reflexes, as well as a change in their shape and intensity, indicates the process of phase transformations as a result of annealing.

During analysis of the initial sample, it was found that after mechanochemical grinding, according to X-ray phase analysis, the structure of the initial composite is a mixture of two phases: cubic neodymium oxide (Nd₂O₃) and a rhomboid phase of hematite (Fe₂O₃) in a content close to 1:1. At the same time, the position and shape of diffraction reflexes for both phases is characteristic of highly disordered structures with a high content of an amorphous-like fraction. This type of structure, characteristic of an interstitial or substitutional solid solution, is most likely for the selected synthesis conditions, and the high content of the amorphous fraction is caused by the grinding processes and partial destruction of phases during the synthesis.

The most characteristic method for increasing the crystallinity degree, as well as for the formation of spinel structures of the NdFeO₃ type, is the method of thermal annealing, which makes it possible not only to increase the degree of structural ordering, but also to initiate phase transformation processes. Analysis of the obtained diffraction patterns of samples under study annealed at a temperature of 600-1000°C showed that with an increase in temperature, the main changes in the diffraction patterns are associated with the appearance of new reflections at $2\theta=24-26^\circ$ and $2\theta=33-36^\circ$, the intensity of which increases with an increase in temperature. At the same time, the main peak at $2\theta=33-34^\circ$ is shifted and divided into two maxima characteristic of the NdFeO₃ and Fe₂O₃ phases. At an annealing temperature of 600°C, the onset of the formation of the NdFeO₃ phase with the partial displacement of the Nd₂O₃ phase is observed (see Figure 3). An increase in the annealing temperature to 800°C leads to the complete transformation of the Nd₂O₃ phase

into the NdFeO_3 phase, with the presence of the Fe_2O_3 phase, the content of which is no more than 25%. This transformation occurs due to the fact that under the influence of temperature, an increase in thermal vibrations of the lattice, as well as annealing of point defects, leads to the processes of the incorporation of iron into the lattice sites of Nd_2O_3 with the subsequent formation of the orthorhombic phase NdFeO_3 of the spatial system $\text{Pnma}(62)$. The complete formation of the NdFeO_3 phase occurs at a temperature of 1000°C , with a small content of the hematite phase (no more than 11%). As is known from the literature [26,27], the hematite phase is re-

sistant to annealing in the temperature range $600\text{-}1000^\circ\text{C}$, which is characterized by the processes of thermal annealing of point defects and vacancies. In this range of annealing temperatures, the hematite structure is ordered and becomes stable. In this case, the NdFeO_3 phase in its structural formula is close to the hematite phase, with only one difference, which consists in the substitution of a neodymium atom for an iron atom. Therefore, the observed structural ordering, characterized not only by a change in the shape of the diffraction lines, but also by a decrease in amorphous inclusions, indicates the annealing of defects and the formation of stable phases.

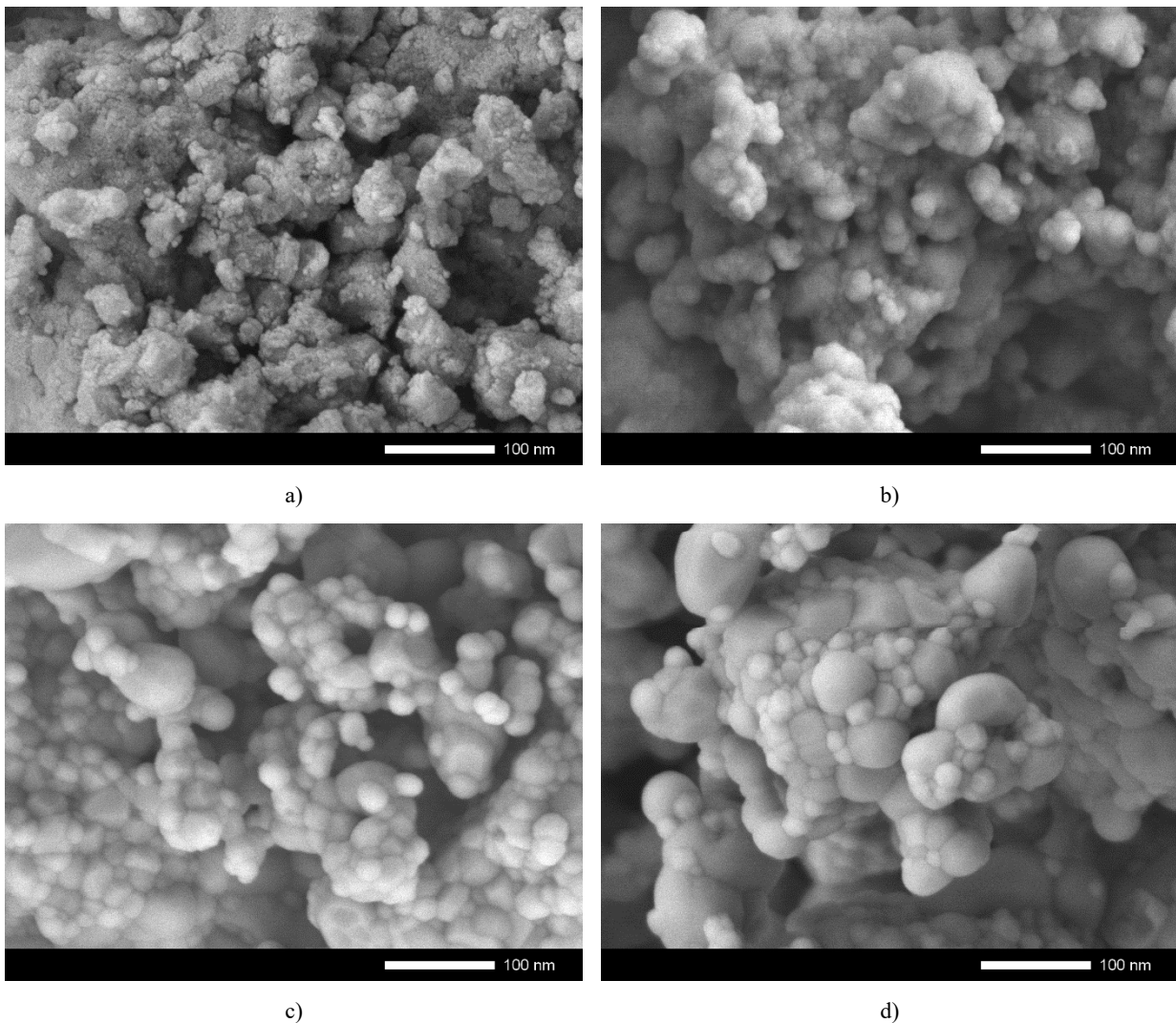


Figure 1 – SEM images of the studied nanocomposites: a) Pristine sample; b) 600°C ; c) 800°C ; d) 1000°C

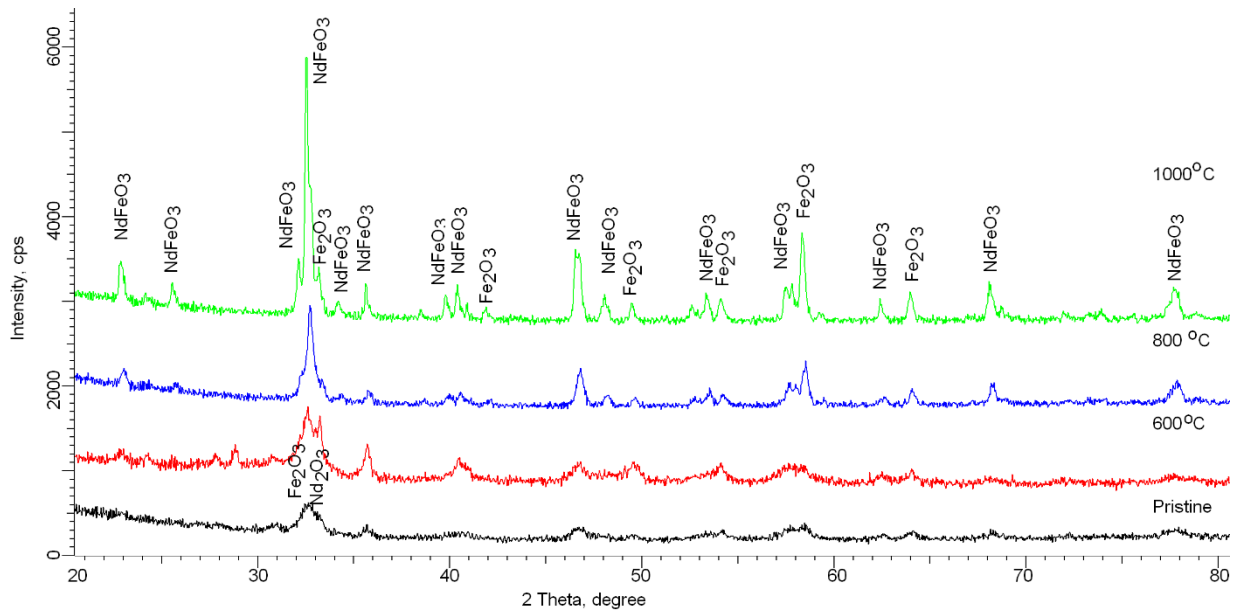


Figure 2 – X-ray diffraction patterns of the studied nanocomposites depending on annealing temperature

Figure 3 shows the results of changes in the phase diagram of nanocomposites calculated on the basis of the obtained X-ray data. According

to this diagram, it can be seen that the main process of phase transformations can be written as follows:

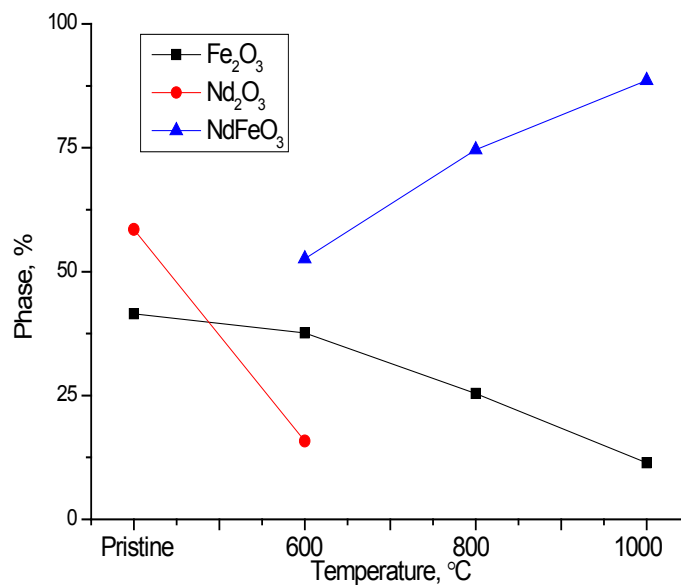
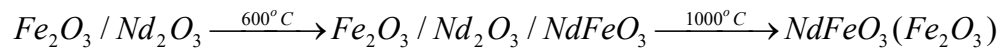


Figure 3 – Dynamics of phase transformations as a result of thermal annealing

Structural parameters such as crystal lattice parameters, crystallite size, dislocation density and degree of crystallinity were calculated on the basis of

the obtained X-ray diffraction patterns. The results of these crystallographic characteristics are presented in Table 1.

Table 1 – Crystallographic data

Parameter	Phase	Pristine	600°C	800°C	1000°C
Crystal lattice parameter, Å	Fe_2O_3	a=5.04646, c=13.78934	a=5.02568, c=13.72175	a=5.00498, c=13.65987	a=4.99615, c=13.63564
	Nd_2O_3	a=11.03509	a=10.99398	-	-
	NdFeO_3	-	a=5.59723, b=7.74027, c=5.43482	a=5.57638, b=7.70840, c=5.41883	a=5.57638, b=7.68573, c=5.40927
Crystallite size, nm		10.6±1.1	12.9±1.4	25.4±2.2	44.9±3.1
Dislocation density, 10^{10} cm^{-2}		8.89	6.01	1.55	0.49
Crystallinity degree, %		74.3	76.1	84.7	87.9

From the data presented in Table 1, it can be seen that thermal annealing leads not only to the processes of phase transformations, but also to the ordering of the structure due to a decrease in dislocation density, an increase in the size of crystallites, as well as an increase in the crystallinity degree, which indicates a decrease in amorphous and disordered inclusions in the structure of annealed composites.

Conclusion

The paper presents the results of the study of the dynamics of phase transformations and changes in the morphological and structural features of the synthesized $\text{Fe}_2\text{O}_3\text{-Nd}_2\text{O}_3 \rightarrow \text{NdFeO}_3/\text{Fe}_2\text{O}_3$ nanocomposites as a result of thermal annealing. Scanning electron microscopy and X-ray phase analysis were used as methods for characterization. During experiments carried out, it was found that the formation of the NdFeO_3 phase begins at a temperature of 600°C, followed by ordering and dominance at higher temperatures. It has been determined that the formation

of the NdFeO_3 phase leads to an increase in the degree of structural ordering, a decrease in amorphous inclusions, and an increase in the grain size. Analysis of the morphological features showed that an increase in the annealing temperature leads not only to an increase in the grain size with the formation of large agglomerates of a dendritic structure, but also to a significant ordering of the grains, which indicates an increase in their density.

The obtained data on the dynamics of phase transformations can be used further as a basis for the development of a technology for the production of magnetic nanocomposites on an industrial scale.

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Conflicts of Interest

The authors declare no conflict of interest.

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